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Total solids determination in dairy products by microwave oven technique

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Abstract

The microwave oven technique is a reliable rapid alternative to classical air-drying in an oven for the determination of the total solids content in high moisture content dairy products. Microwave oven drying has the advantage of a selective heating of water which, combined with the short drying time, results in less lactose hydration and browning. The reduction of weight loss caused by degradation results in improved repeatability and reproducibility. This allows use of this technique as a rapid near-line technique, performed by operational staff, for release of intermediate or final product. The measurement is fast with a time of analysis of less than 5 min. Today's instrumentation is user-friendly and sample preparation is generally straightforward. As for all total solids determinations, a representative sampling is required and sometimes the sample needs to be homogenized. In some high fat products, dilution helps to improve the penetration of the product into the sample pad and afterwards favours water release during the drying process.

Keywords: Water content; Microwave drying; Rapid method; Dairy products

1. Introduction

1.1. General

The total solids content of a product is a key parameter in the food industry. It can be part of the determination of the final product composition. The water content of a product is often regulated by legislation and might strongly influence the product's stability. During food processing, the total solids content often affects final product quality. Most processing steps are influenced by the physico-chemical characteristics of the processed media. Reaction rates in the food matrices are influenced by the ingredient concentration, the effectiveness of thermal treatments is related to the heat transfer rates into the matrices and physical transformations, such as homogenization, are affected by the viscosity of the matrices. Great efforts are therefore taken to optimize the control of this parameter.

The other important consideration is the response time. A short analysis time allows rapid liberation of a batch of intermediate product and can lead to a better utilization of production installations. Despite all efforts engaged in developing indirect rapid methods to determine the total solids content, a more rapid direct technique is required for a large number of applications, especially at production sites with a range of different products where performing a limited number of accurate and direct measurements has clear advantages.

The microwave oven technique is the most prominent direct rapid method. Due to selective heating of the water molecules by microwave radiation, evaporation takes place extremely rapidly. This results in a very short analysis time, which limits the exposure of the sample to heat and, therefore, to weight loss due to degradation reactions. This is especially important for products containing sugars. Lactose is present in almost all dairy products and other sugars, such as glucose or sucrose are often added to related products, such as yogurt or ice cream. New microwave ovens adjust the microwave energy by controlling either the product temperature or the weight loss curve in order to minimize degradation. This study is aimed at evaluating how repeatability and reproducibility of the microwave oven method is affected by these improvements compared to earlier studies.

1.2. Total solids determination by different techniques

The classical procedure of determining total solids content is based on weight loss: the sample is weighed,

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then placed in an air-drying oven and warmed to a defined temperature in order to evaporate the water. In most cases the sample is mixed with seasand in order to facilitate evaporation by increasing the surface area and, in the case of high fat products, to avoid the retention of water by a surface layer. After a defined waiting time, the sample has lost the water and is weighed again. The measured weight difference is attributed to the water and, therefore, the total solids content is determined indirectly. This is based on several assumptions and precautions. The first assumption is that only water molecules are lost, and secondly that all the water present is released by the procedure. Practice has shown that neither assumption is completely valid. Dependent on the product, other volatile ingredients, such as aroma compounds, can be released. In most food products, the amount of these compounds is relatively small compared to the water content. The temperature chosen for the drying process also influences the losses. It is important to select an appropriate drying temperature, which avoids decomposition of samples. Products with high carbohydrate and protein contents are susceptible to degradation related to Maillard reactions. These reactions can therefore lead to an artificially lower total solids content. Often, reduced pressure is applied in order to reduce the drying temperature. However, the complete evaporation of water is still an issue for the total solids determination.

In industry the total solids content is often determined by indirect methods, such as refractometry, infrared spectroscopy, density or microwave spectroscopy. All these methods require calibration and a reliable reference method. The validity of the calibrations is often restricted to a relatively small range of total solids content and a single product. Modifications in the physicochemical aspect of the product can additionally affect the measurement.

1.3. Microwave oven for total solids determination

Literature about determination of total solids content by microwave oven-drying is quite limited, as it is not an official reference method. Nevertheless, several studies were undertaken to investigate the feasibility of using the technique as a rapid alternative method. In a collaborative study, total solids in processed tomato products were determined by microwave oven-drying, employing the CEM AVC-MP analyzer (Chin, Kimball, Hung, & Allen, 1985). The total solids content in this study varied between 6.2 and 40.3 wt.% and the results were compared with total solids content determined by the vacuum oven method at 70 °C. The study, including fourteen laboratories, showed no difference between the techniques. The results showed additionally good repeatability and reproducibility for the whole range of total solids. Another collaborative study, involving eight laboratories, determined the total solids content in tomato pulp (Wang, 1987) and concluded that the technique is a valuable rapid alternative to the vacuum oven method. The CEM analyzer AVC-MP was also tested for rapid moisture determination in flour (Davis & Lai, 1984). After optimization of the method, the microwave method showed consistently lower results than the standard air-oven method. Despite the 0.5 wt.% difference, the authors concluded that the microwave method is a good method for rapid moisture determination in flour, due to the consistency of the results. Another study was organized using microwave ovens as a rapid and inexpensive method for total solids determination in fluid dairy products (Dzurec & Baptie, 1989). The study compared commercially available microwave ovens with the air oven method. Results were comparable but it was found that training of the technicians running the technique is of key importance to achieve consistent results. The possibility of using the CEM AVC 80 as a rapid alternative method to,the AOAC air-oven method in liquid milk products was studied (Makarchuck, Hill, & Szijarto, 1991). The AOAC method is generally used for the calibration of infrared analyzers for total solids content. The authors preferred the air-oven method due to better instrument repeatability. A study on the microwave drying was performed (Oomah & Mazza, 1992) for the moisture determination in flax, canola and yellow mustard seeds, using a standard home microwave oven. The results showed the capability of microwave oven-drying as a rapid alternative technique for oilseeds.

2. Materials and methods

2.1. Microwave oven

The measurements in this study were performed using the CEM Smart System *5* instrument. The system delivers 250 W of microwave energy when programmed for 100% power. The voltage of the incoming electrical service is measured and the microwave power is adjusted by the power control system in order to normalize the power and to provide repeatable power conditions. The basic system components are a microwave drying chamber, an electronic balance, an infrared temperature controller and a microprocessor. The infrared temperature measurement permits automatic adjustment of the microwave power as a function of a user-defined sample temperature. This avoids over-heating of the sample and degradation of the product. The 50-g analytical balance has a sensitivity of ± 0.1 mg.

Proper sampling and sample preparation are critical for accuracy and precision. It is therefore important to obtain representative and homogeneous samples. Heterogeneous samples, such as dairy products containing fruit

pieces, were homogenized using a high-speed homogenizer and all samples were stirred well before being placed on the sample pads. The glass fibre sample pads are a key element of the microwave drying procedure as they help to increase the surface area of the sample and facilitate water release. The instrument allows sample weights between 1 and 15 g. For concentrated samples a smaller sample weight is advised whereas samples with low total solids content should be determined using a larger weight. For the samples in our studies, between 2 and 3 g were measured. Some samples were diluted with distilled water prior to the measurement, in order to improve the spreadabiity of the sample. It is of key importance to distribute the sample well over the pad and allow the sample to enter well into the pad material. We used a second sample pad to cover the first pad which allowed us to better absorb the sample. Additionally, crust formation is reduced to a minimum by this procedure. It is important to guarantee the presence of sample in the middle of the pad as the temperature of the sample is controlled at that point. Some samples containing strong microwave absorbers, such as carbohydrates, might require a reduction of the microwave energy to prevent overheating and related degradation.

Our measurement procedures were developed with the aim of matching the air-drying oven results as closely as possible and to optimize the repeatability of the determinations. Preference was given to repeatability, as it is known that, in some cases, a bias might occur, due to the technical differences of the drying techniques. When used in the production environment, it is more important to guarantee repeatability and reproducibility as a bias correction can easily be made. The product-related parameters which cause this bias must be studied to ensure that the bias stays constant over time.

The measurement time is generally between 5 and 10 min which includes some time for cooling the sample before the final weighing. For most of the dairy products, measurement times of around 5 min are most common. The principal water loss occurs during the first two min, as shown in Fig. 1, for a standard pasteurised milk with a total solids content of around 12 wt.%. The drying time depends on the water content, the chosen drying conditions and sometimes also on the composition of the product. The key elements for the short measurement time are preferential heating of the water molecules by the microwave energy and the large surface or evaporation provided by the glass-fibre filter pad.

2.2. Oven-drying

Reference values were obtained using the air-drying oven. The same precautions regarding sampling and sample preparation apply as for the microwave oven determinations including the high-speed homogenization for products containing pieces. The drying procedure used corresponds to IDF norms 15B and 21B. Samples of approximately 3.0 g are mixed with 25 g pre-dried sand and dried in a well-controlled drying oven for 4 h at $102 \pm 2\ ^{\circ}C$ using dishes and lids of pure nickel. The dishes of 75 mm diameter and 25 mm height are pre-dried with

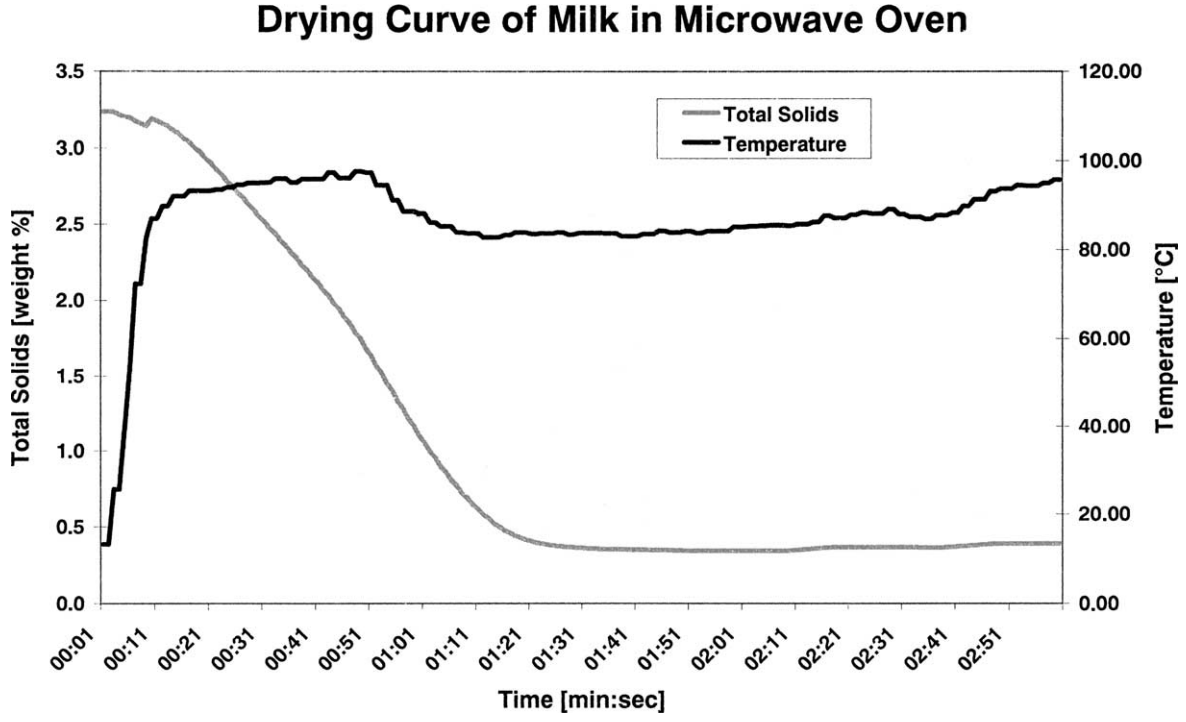

Fig. 1.

25 g sand for at least 1 h at 102±2 °C. After cooling for 45 min in the desiccator, they are weighed. About 3 g of product is added and well mixed with the sand using a glass rod. After drying for 4 h at 102±2 °C, the dish is cooled for 45 min in a desiccator and weighed. The total solids or dry matter content is then calculated. The number of samples in the oven is limited in order to allow proper drying. The used air-drying oven is placed under controlled ambient temperature and relative humidity in order to limit external influences on the results.

2.3. Tested dairy products

2.3.1. Sweetened condensed milk

Sweetened condensed milk is concentrated milk with added sugar. In order to obtain very small sugar crystals, seeding lactose is added during production. The total solids content is quite elevated with values often higher than 70 wt.%. The sample set consisted of 13 sweetened condensed milks produced in the pilot plant for product development purposes. The reference analysis was performed in our laboratories according to the mentioned total solids air-drying oven technique. The product is highly susceptible to degradation, due to high total solids, combined with high sugar and high fat content. To avoid crust formation and related degradation of the lactose, the samples were diluted. When diluting 1 part of sweetened condensed milk with three parts of distilled water, the sample can be dried without any browning occuring. The maximum temperature was chosen as 100 °C and the sample weight between 2 and 3 g.

2.3.2. Non-sweetened condensed milk

Non-sweetened condensed milk is concentrated milk without added sugar. The total solids content, with between 20 and 30 wt.%, is smaller than that of the sweetened condensed milk. Our sample set consisted of eight samples of non-sweetened condensed milk. The reference values are based on the air-drying oven for 4 h at 102 °C. It was not necessary to dilute as the composition allows a good release of the water. The maximum temperature was chosen as 102 °C and the used sample weight between 2 and 3 g.

2.3.3. Ice cream premixes

Ice cream premixes are the intermediate product of ice cream production before freezing. The total solids content can vary between around 25 and 50 wt.%, depending on the final use and the type of formula. Overall, 34 samples were available within our sample set out of normal production from the pilot-plant. The reference analysis was performed in two different laboratories, using the air-drying oven technique. The sample sets were therefore split for some statistical calculations. Especially the high total solids samples tend to show degradation of the sample, similar to the behaviour of the sweetened condensed milk. Despite that risk, we decided to avoid dilution in order to keep the sample preparation as simple as possible. The maximum temperature was chosen as 100 °C and the sample weight between 2 and 3 g. No dilution was performed.

2.3.4. Yogurt and quark

Yogurt and quark are dairy products with total solids contents between 15 and 30 wt.%. This study primarily focused on repeatability for the measurements of yogurt and quark, with a limited number of samples. During this study no reference analysis by air-drying oven was conducted. The analyzed samples consisted of a traditional yogurt, two stirred yogurts and a low fat quark. The maximum temperature was chosen as 100 °C and the sample weight between 2 and 3 g. The yogurt samples containing fruit pieces were homogenized with a laboratory mixer but not diluted.

2.3.5. Other dairy products

In this part of the study, we analyzed a number of dairy products with varying proportions of protein, fat, carbohydrate and total solids. The sample set consisted of 27 dairy products produced in the pilot plants for product development purposes. The reference analysis was performed in the laboratories according to the mentioned air-drying oven technique. The total solids content ranged between 12 and 27 wt.%. The maximum temperature for the microwave oven was chosen as 100 °C and the sample weight between 2 and 3 g. No dilution was performed.

3. Results and discussion

3.1. Sweetened condensed milk

The results of the microwave oven and the air-drying oven for 13 samples of sweetened condensed milk are shown in Table 1. The table shows the mean of two repetitions. The standard deviation of repeatability was better than 0.20 wt.%. This results in a coefficient of variation of less than 0.30 wt.% for all samples. Linear regression on the two sample sets showed R^2 of 0.987. This is a good result, taking the small range of total solids content into account. A significant bias of around 1.0 wt.% was identified. This bias is the largest identified during this study, caused by the high total solids and high fat content of the product. Despite the dilution step, sweetened condensed milk seems to produce a crust of fat and protein which can also close some of the pores present in the filter paper. This illustrates the importance of having an increased surface area for the total solids determination. In the case of the air-drying oven the sand perfoms this function. The sample pad

Table 1
Comparison of microwave oven and air oven for sweetened condensed milk

	CEM microwave oven	Oven	Difference CEM–oven
Sample 1	73.96	72.74	1.22
Sample 2	73.72	72.30	1.42
Sample 3	74.50	73.50	1.00
Sample 4	74.32	73.42	0.90
Sample 5	73.34	72.08	1.26
Sample 6	75.59	74.60	0.99
Sample 7	74.06	72.88	1.18
Sample 8	73.52	72.32	1.20
Sample 9	74.60	73.26	1.34
Sample 10	74.5	173.32	1.19
Sample 11	5.69	74.69	1.00
Sample 12	75.25	74.19	1.06
Sample 13	74.31	73.35	0.96
S.D.	0.74	0.84	0.16
Average	74.41	73.28	1.13
Minimum	73.34	72.08	0.90
Maximum	75.69	74.69	1.42

and its structure is the crucial element for the microwave oven. It also was important to distribute the sample well over the pad. Improvements in repeatability and the stabilisation of the determined bias are parameters which indicate good and stable drying conditions. Under the chosen conditions, no deterioration, e.g. browning of the sample, was noted. This is principally due to the restriction of the sample temperature, controlled by the infrared thermometer. When the temperature was higher than the chosen 100 °C, degradation took place. Typical signs were caramelisation of the sugar, browning and, as a result, a reduction of the repeatability. This observation was also valid for the other products measured in this study.

3.2. Non-sweetened condensed milk

The results of the microwave oven and the air-drying oven for the eight samples of non-sweetened condensed milk are shown in Table 2. Each sample was measured by the microwave oven 10 times. The standard deviation of these 10 repetitive measurements is better than 0.05 weight%. This results in a coefficient of variation of less than 0.2 wt.% for all samples. No significant bias was identified when comparing the results of both methods. The standard deviation of differences calculates as 0.08 wt.%. Linear regression on the two sample sets showed an R^2 of 0.999. The statistical results suggest that the microwave oven technique is an equivalent technique to the air-drying oven.

3.3. Ice cream premixes

The results of the microwave oven and the air-drying oven for 22 samples of ice cream premixes are shown in Table 3. The Table shows the mean of two repetitions. The standard deviation of repeatability was determined as better than 0.15 wt.%. Repeatability tends to be better for samples with lower total solids and total fat contents. For these samples, a standard deviation of repeatability better than 0.10 wt.% was found. Repeatability for high total solids, high fat samples can be improved by dilution, of the sample prior to measurement. These measurements result in a coefficient of variation of less than 0.3 wt.% for all samples. A bias was identified when comparing the results of both methods. This bias increases with increasing total solids and total fat contents. When using this method in a production environment the products need to be classified according to their composition and, for each group, the bias has to be established. Linear regression on the two sample sets gave R^2 of 0.997.

Table 2
Repeatability data of non-sweetened condensed milk by CEM microwave oven

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
CEM 1	25.02	23.41	21.50	25.22	21.18	23.40	24.07	26.43
CEM 2	25.02	23.41	21.51	25.22	21.14	23.48	24.02	26.40
CEM 3	24.94	23.41	21.55	25.13	21.15	23.45	24.01	26.47
CEM 4	25.02	23.45	21.62	25.13	21.14	23.46	24.01	26.42
CEM 5	25.04	23.43	21.56	25.12	21.14	23.41	24.09	26.47
CEM 6	25.08	23.53	21.61	25.16	21.23	23.42	23.99	25.50
CEM 7	25.02	23.49	21.59	25.14	21.21	23.44	23.97	26.49
CEM 8	25.05	23.48	21.50	25.14	21.15	23.43	24.06	26.49
CEM 9	25.03	23.45	21.57	25.14	21.09	23.50	24.04	26.46
CEM 10	25.01	23.38	21.58	25.12	21.18	23.41	24.01	26.47
Average	25.02	23.44	21.56	25.15	21.16	23.44	24.03	26.46
S.D.(r)	0.046	0.045	0.041	0.038	0.040	0.033	0.037	0.033
cv%	0.18	0.19	0.19	0.15	0.180	0.14	0.15	0.12
Oven 4 h. 102 °C	24.97	23.36	21.52	25.11	20.99	23.23	23.88	26.48

Table 3
Comparison of microwave oven and air oven drying for ice cream premixes

Samples	Total solids (wt.%)		
	CEM microwave oven	Oven	Difference CEM–oven
Ice cream 1	52.46	51.4	1.06
Ice cream 2	52.42	50.85	1.57
Ice cream 3	37.06	36.95	0.11
Ice cream 4	46.65	45.27	1.38
Ice cream 5	51.43	50.29	1.14
Ice cream 6	39.31	39.17	0.14
Ice cream 7	50.93	50.43	0.50
Ice cream 8	47.70	46.83	0.87
Ice cream 9	35.84	35.68	0.16
Ice cream 10	36.45	36.28	0.16
Ice cream 11	36.23	35.74	0.49
Ice cream 12	36.22	35.72	0.50
Ice cream 13	29.58	29.52	0.06
Ice cream 14	34.60	34.21	0.38
Ice cream 15	35.48	35.14	0.34
Ice cream 16	34.33	33.95	0.38
Ice cream 17	24.22	23.48	0.74
Ice cream 18	40.52	40.14	0.38
Ice cream 19	41.40	41.38	0.02
Ice cream 20	35.16	35.18	−0.02
Ice cream 21	36.44	37.09	−0.65
Ice cream 22	40.60	39.83	0.77
S.D.	7.58	7.29	0.51
Minimum	24.22	23.48	−0.65
Maximum	52.46	51.40	1.57
Average	39.77	39.30	0.47

Table 4
Comparison of microwave oven and air oven drying for divers dairy products

Samples	Total solids (wt.%)		
	Oven	CEM microwave oven	Difference CEM–oven
Sample 1	25.36	25.58	0.22
Sample 2	25.53	25.63	0.10
Sample 3	24.14	24.54	0.40
Sample 4	14.70	14.78	0.08
Sample 5	23.95	24.32	0.37
Sample 6	25.44	25.68	0.24
Sample 7	26.24	26.50	0.26
Sample 8	25.60	25.77	0.17
Sample 9	24.06	24.33	0.27
Sample 10	24.14	24.39	0.25
Sample 11	25.83	26.24	0.41
Sample 12	24.45	24.60	0.15
Sample 13	19.40	19.65	0.25
Sample 14	24.30	24.70	0.40
Sample 15	25.24	25.45	0.21
Sample 16	25.17	25.49	0.32
Sample 17	18.41	18.44	0.03
Sample 18	18.30	18.33	0.03
Sample 19	19.11	19.12	0.01
Sample 20	19.29	19.29	0.00
Sample 21	12.50	12.43	−0.07
Sample 22	23.95	24.10	0.15
Sample 23	12.30	12.40	0.10
Sample 24	20.06	20.28	0.22
Sample 25	24.69	25.13	0.44
Sample 26	25.87	26.10	0.23
Sample 27	25.18	25.50	0.32
S.D.	4.18	427	0.14
Minimum	12.30	12.40	−0.07
Maximum	26.24	26.50	0.44
Average	2.34	22.55	0.21

3.4. Yogurt and quark

The results for yogurt and quark are shown in Table 4. The standard deviations of six repetitive measurements of a standard yogurt, a flavoured yogurt, a fruit yogurt and alow fat quark were 4.18 and 4.27. Measurement normally takes less than 5 min and the instrument allows total solids determination near to the production line. Therefore rapid decisions for releasing intermediate or final products can be made. The required operator skill is relatively small and, after a short training, operational staff can be trained to perform this type of analysis.

The performance of the new microwave oven was significantly improved compared to studies done with earlier models in the 80's and 90's. Controlling either the sample temperature or the drying process allows improvement of repeatability and reproducibility. For some samples with high total solids content, combined especially with high total fat content, a dilution step is necessary. For some products, the method performance is sufficiently good to be used for calibration of indirect methods, such as refractometry or infrared spectroscopy. Despite the excellent precision, a bias is sometimes identified compared to the air oven technique. Even if the same weight loss as conventional drying is achieved, it cannot be assumed that the composition of the volatiles is exactly the same. This is the weakness of all comparisons between drying techniques. The drying mechanisms of the microwave oven and the air-drying oven are obviously very different and it is, therefore, not surprising that we have observed a bias, especially for high total solids, high fat products. Further studies are needed to further clarify to what degree samples are degraded or moisture is still not released from the matrix.

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